

FORM PTO-1390 (Modified) (REV 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER <b>71247-0003</b>	
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371				U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR <b>10/069103</b>	
INTERNATIONAL APPLICATION NO. <b>PCT/FR00/02384</b>		INTERNATIONAL FILING DATE <b>25 AUGUST 2000 (25.08.00)</b>		PRIORITY DATE CLAIMED <b>26 AUGUST 1999 (26.08.99)</b>	
TITLE OF INVENTION <b>NON-ETHANOL COMPOSITION COMPRISING HYDROFLUOROETHER</b>					
APPLICANT(S) FOR DO/EO/US <b>Marie-Laure SOUVIE and Gerard BATON</b>					
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:					
<ol style="list-style-type: none"> <li>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li>3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (24) indicated below.</li> <li>4. <input type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31).</li> <li>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371 (c) (2)) <ol style="list-style-type: none"> <li>a. <input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau).</li> <li>b. <input checked="" type="checkbox"/> has been communicated by the International Bureau.</li> <li>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</li> </ol> </li> <li>6. <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)). <ol style="list-style-type: none"> <li>a. <input checked="" type="checkbox"/> is attached hereto.</li> <li>b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4).</li> </ol> </li> <li>7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3)) <ol style="list-style-type: none"> <li>a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau).</li> <li>b. <input type="checkbox"/> have been communicated by the International Bureau.</li> <li>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</li> <li>d. <input checked="" type="checkbox"/> have not been made and will not be made.</li> </ol> </li> <li>8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</li> <li>9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).</li> <li>10. <input type="checkbox"/> An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).</li> <li>11. <input type="checkbox"/> A copy of the International Preliminary Examination Report (PCT/IPEA/409).</li> <li>12. <input type="checkbox"/> A copy of the International Search Report (PCT/ISA/210).</li> </ol> <p><b>Items 13 to 20 below concern document(s) or information included:</b></p> <ol style="list-style-type: none"> <li>13. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</li> <li>14. <input checked="" type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</li> <li>15. <input checked="" type="checkbox"/> A <b>FIRST</b> preliminary amendment.</li> <li>16. <input type="checkbox"/> A <b>SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.</li> <li>17. <input type="checkbox"/> A substitute specification.</li> <li>18. <input type="checkbox"/> A change of power of attorney and/or address letter.</li> <li>19. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.</li> <li>20. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4).</li> <li>21. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).</li> <li>22. <input type="checkbox"/> Certificate of Mailing by Express Mail</li> <li>23. <input type="checkbox"/> Other items or information:</li> </ol>					

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 101.11) <b>10/069103</b>		INTERNATIONAL APPLICATION NO. <b>PCT/FR00/02384</b>		ATTORNEY'S DOCKET NUMBER <b>71247-0003</b>	
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24. The following fees are submitted:

<b>BASIC NATIONAL FEE ( 37 CFR 1.492 (a) (1) - (5)) :</b>			<b>CALCULATIONS PTO USE ONLY</b>	
<input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... <b>\$1040.00</b> <input checked="" type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... <b>\$890.00</b> <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... <b>\$740.00</b> <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... <b>\$710.00</b> <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... <b>\$100.00</b>				
<b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>				<b>\$890.00</b>
Surcharge of \$130.00 for furnishing the oath or declaration later than _____ months from the earliest claimed priority date (37 CFR 1.492 (e)). <input type="checkbox"/> 20 <input type="checkbox"/> 30				<b>\$0.00</b>
<b>CLAIMS</b>	<b>NUMBER FILED</b>	<b>NUMBER EXTRA</b>	<b>RATE</b>	
Total claims	34 - 20 =	14	x \$18.00	<b>\$252.00</b>
Independent claims	4 - 3 =	1	x \$84.00	<b>\$84.00</b>
Multiple Dependent Claims (check if applicable). <input type="checkbox"/>				<b>\$0.00</b>
<b>TOTAL OF ABOVE CALCULATIONS =</b>				<b>\$1,226.00</b>
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27). The fees indicated above are reduced by 1/2.				<b>\$0.00</b>
<b>SUBTOTAL =</b>				<b>\$1,226.00</b>
Processing fee of \$130.00 for furnishing the English translation later than _____ months from the earliest claimed priority date (37 CFR 1.492 (f)). <input type="checkbox"/> 20 <input type="checkbox"/> 30			+	<b>\$0.00</b>
<b>TOTAL NATIONAL FEE =</b>				<b>\$1,226.00</b>
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable). <input checked="" type="checkbox"/>				<b>\$40.00</b>
<b>TOTAL FEES ENCLOSED =</b>				<b>\$1,266.00</b>
			Amount to be:	\$
			refunded	\$
			charged	\$

a. ☐ A check in the amount of \_\_\_\_\_ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 50-1088 in the amount of \$1,266.00 to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 50-1088. ~~A duplicate copy of this sheet is enclosed.~~

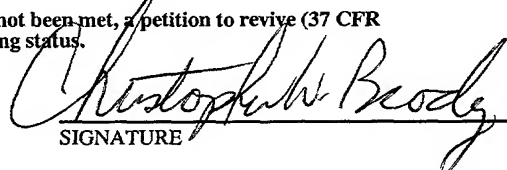
d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

**NOTE:** Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

**SEND ALL CORRESPONDENCE TO:**

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 SIGNATURE

**Christopher W. Brody**  
 NAME

**33,613**  
 REGISTRATION NUMBER

**February 22, 2002**  
 DATE

PATENTIN THE UNITED STATES PATENT & TRADEMARK OFFICE

Applicants: SOUVIE, Marie-Laure et al.  
International Application No.: PCT/FR00/02384  
International Filing Date: August 25, 2000  
Serial Number: To be assigned  
U.S. Filing Date: February 22, 2002  
For : Non-ethanol Composition Comprising Hydrofluoroether.

PRELIMINARY AMENDMENT

Assistant Commissioner of Patents  
Washington, DC 20231

Sir :

Before the calculation of the filing fee, please amend the above identified patent application as follows:

IN THE CLAIMS

Please cancel claims 1 - 23 and add claims 24-57 as indicated below:

24. An ethanol-free or alcohol-free composition comprising a hydrofluoro ether component and at least one polyacid ester.
25. The composition of claim 24, wherein the hydrofluoro ether component is a perfluorinated component of the general formula  $C_nH_mF_p-O-C_xH_yF_z$ , in which n is a number ranging from 1 to 12, m is a number ranging from 0 to 25, p is a number ranging from 0 to 11,  $m + p = 2n + 1$ , x is a number from 1 to 12, y is a number from 0 to 25, x is a number from 0 to 11 and  $y + z = 2x + 1$ , and in which m and y may not be equal to 0 simultaneously and p and z may not be equal to 0 simultaneously.
26. The composition of claim 24, wherein the hydrofluoro ether component is selected from the group consisting of methoxynonafluorobutane, ethoxynonafluorobutane, propoxyundecafluoropentane and methoxyheptafluoropropane.
27. The composition of claim 24, wherein the polyacid ester is an ester of a hydroxylated or non-hydroxylated polyacid esterified with a saturated or unsaturated, linear or branched alcohol having between 1 and 30 carbon atoms.
28. The composition of claim 24, wherein the polyacid ester is esterified with an alcohol having between 1 and 12 carbon atoms.

29. The composition of claim 24, wherein the polyacid ester is esterified with an alcohol selected from the group consisting of ethanol, isopropanol and 2-ethylhexanol.
30. The composition of claim 24, wherein the polyacid has between 3 and 10 atoms and comprises a linear or branched, saturated or unsaturated carbon chain optionally substituted by at least one substituent selected from the group consisting of hydroxyl, ketone, and hydroxyl substituted by acetyl.
31. The composition of claim 24, wherein the polyacid is totally esterified with a saturated or unsaturated, linear or branched alcohol having between 1 and 30 carbon atoms.
32. The composition of claim 24, wherein the polyacid is a saturated diacid selected from the group consisting of malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid and azelaic acid; a monounsaturated diacid selected from the group consisting of fumaric acid, maleic acid, citraconic acid, itaconic acid and mesaconic acid; a diunsaturated diacid; muconic acid; a monohydroxylated diacid; tartronic acid; malic acid; citramalic acid; a dihydroxylated diacid; dihydroxymaleic; tartaric acid, a tetrahydroxylated diacid; dihydroxytartaric acid; galactaric acid; glucaric acid; a keto diacid; mesoxalic acid; oxalacetic acid; 2-oxoglutaric acid; 3-oxoglutaric acid, a diketo diacid; 2,3-diketoadipic acid, a saturated triacid; tricarballic acid; citric acid, an unsaturated triacid; and aconitic acid.
33. The composition of claim 24, wherein the polyacid is citric acid
34. The composition of claim 24, wherein the polyacid is adipic acid.
35. The composition of claim 24, wherein the polyacid ester is a substantially non-polar ester.
36. The composition of the claim 24, wherein the polyacid ester is selected from the group consisting of triethyl citrate, tri(2-ethylhexyl) citrate, diisopropyl adipate and di(2-ethylhexyl) adipate.
37. The composition of claim 24, wherein the polyacid ester represents from 0.1 to 30% by weight of the hydrofluoro ether component.
38. The composition of claim 24, wherein the polyacid ester represents from 1 to 20% by weight of the hydrofluoro ether component
39. The composition of claim 24, further comprising a complementary component, selected from a second co-solvent and a component for improving the properties of a perfume composition.
40. The composition of claim 39, wherein the co-solvent is a silicone, and the component for improving the properties of a perfume composition, promotes persistence of said composition on the skin and comprises a phthalate
41. The composition of claim 40, wherein said phthalate is diethyl phthalate.

42. The composition of claim 24, further comprising a silicone selected from a volatile silicone, a dimethicone, a cyclomethicone, pentacyclomethicone, and an organotrisiloxane, the silicone representing from 1 to 20% by weight of the composition.

43. An ethanol-free or alcohol-free perfume composition comprising a perfume concentrate, a hydrofluoro ether component and at least one polyacid ester in an amount sufficient to give said composition an essentially clear appearance.

44. The perfume composition of claim 43, wherein the hydrofluoroether component is present in an amount ranging from about 65 to about 85% by weight, based on the weight of the perfume composition.

45. The composition of claim 44, comprising from about 1 to about 20% by weight of polyacid ester, based on the weight of the perfume composition.

46. The composition of claim 43, comprising from about 5 to about 20% by weight of perfume concentrate.

47. The composition of claim 43, formulated as a body lotion comprising a concentration of perfume concentrate in the order of 5% by weight, based on the weight of the composition.

48. The composition of claim 43, formulated as a perfume, the concentration of perfume concentrate being between 10 and 20% by weight of the perfume composition.

49. The composition of claim 43, *wherein the hydrofluoro ether component is a perfluorinated component of the general formula  $C_nH_mF_p-O-C_xH_yF_z$  in which n is a number ranging from 1 to 12, m is a number ranging from 0 to 25, p is a number ranging from 0 to 11,  $m + p = 2n + 1$ , x is a number from 1 to 12, y is a number from 0 to 25, x is a number from 0 to 11 and  $y + z = 2x + 1$ , and in which m and y may not be equal to 0 simultaneously and p and z may not be equal to 0 simultaneously.*

50. The composition of claim 43, comprising an additional component, selected from a second co-solvent, and a component for improving the properties of the perfume composition,

51. The composition of claim 50, wherein the co-solvent is a silicone and the component for improving the properties of the perfume composition promotes the persistence of said composition on the skin, and comprises a phthalate.

52. The composition of claim 43, further comprising up to 2% by weight, based on the composition, of at least one additional additive including a UV filter, an antioxidant or a dye.

53. A method of solubilizing a perfume concentrate comprising admixing the perfume concentrate with a hydrofluoroether component and a polyacid ester.

54. The method of claim 53, wherein the hydrofluoro ether component is a perfluorinated component of the general formula  $C_nH_mF_p-O-C_xH_yF_z$ , in which n is a number ranging from 1 to 12, m is a number ranging from 0 to 25, p is a number ranging from 0 to 11,  $m + p = 2n + 1$ , x is a number from 1 to 12, y is a number from 0 to 25, x is a

number from 0 to 11 and  $y + z = 2x + 1$ , and in which m and y may not be equal to 0 simultaneously and p and z may not be equal to 0 simultaneously.

55. The method of claim 53, further comprising preparing a perfume composition selected from a perfume and a body lotion.

56. An ethanol-free or alcohol-free perfume composition, comprising a perfume concentrate, hydrofluoro ether component selected from the group consisting of methoxynonafluorobutane, ethoxynonafluorobutane, propoxyundecafluoropentane and methoxyheptafluoropropane, and a polyacid ester selected from the group consisting of triethyl citrate, tri(2-ethylhexyl) citrate, diisopropyl adipate and di(2-ethylhexyl) adipate.

57. The composition of claim 56, further comprising a silicone selected from a volatile silicone, a dimethicone, a cyclomethicone, pentacyclomethicone, and an organotrisiloxane, the silicone representing from 1 to 20% by weight of the composition.


#### REMARKS

Claims 1 through 23 have been cancelled and claims 24-57 are submitted for examination. No new matter has been added.

Please charge any fee deficiency or credit any overpayment to Deposit Account No. 50-1088.

Respectfully submitted,

CLARK & BRODY



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Docket No.: 71247-0003  
Date: February 22, 2002

Non-ethanolic composition comprising a hydrofluoroether

The invention relates essentially to a non-ethanolic composition comprising a perfluorinated hydrofluoro ether and at least one co-solvent, other than water and ethanol, comprising a polyacid ester, and to its use in perfume compositions.

5 Such perfume compositions are preferably ethanol-free perfume compositions of which all the components are miscible with one another to give the composition the appearance of a clear liquid.

10 In perfume products (perfume, toilet water, etc.), the presence of alcohol (ethanol), which is used mainly as a solubilizer for the perfume concentrate, presents a number of problems well known to those skilled in the art. It is for this reason that research has been carried out for many years on perfume products which avoid the addition of alcohol by replacing it with other solubilizers.

15 As examples, reference may be made to the documents WO 99/18925 and US 5,468,725, which describe alcohol-free perfume compositions and respectively use silicones as solubilizer and the microemulsion technique.

One of the general problems which arise is the olfactory preservation of the perfume concentrate composition and especially the olfactory neutrality of the solubilizers used.

20 Furthermore, the document WO 99/11225 discloses cosmetic preparations in which the essential ingredients are at least 1% of hydrofluoro ether for the purpose of improving the tolerability of these compositions on the skin and improving the feel of the cosmetic product.

25 Also, the document WO 99/26600 discloses the use of perfluorinated hydrofluoro ethers as agents for dissolving aromatic compounds in the preparation of a cosmetic composition. On page 3, lines 21 to 25, said document envisages in general terms the possibility of adding at least one co-solvent, which is indicated as preferably being selected from the group comprising ethanol and water, i.e. in practice an aqueous-alcoholic mixture. The Examples given in said document all relate to the exclusive use of a perfluoro ether for solubilizing essential oils.

30 The hydroperfluoro ethers mentioned are methoxynonafluorobutane, abbreviated to MNFB, in Examples 1 and 2, nonafluorobutane, abbreviated to ENFB, in Examples 3 to 8, and also propoxyundecafluoropentane.

A specific problem exists with perfume compositions, namely the necessary

solubility of all the constituents of such a perfume composition. A principal component is made up of a perfume concentrate containing essential oils together with various components such as emulsifiers or surfactants, fatty esters or cellulose derivatives, together with other components well known to those skilled in the art.

5 In the context of experiments performed with a view to obtaining ethanol-free or alcohol-free compositions, the Applicant performed experiments aimed at verifying whether perfluorinated hydrofluoro ethers were capable on their own of totally solubilizing such perfume concentrates in order to prepare perfume compositions, these perfume concentrates being commercially available and  
10 marketed by companies specialized in their manufacture, such as GIVAUDAN, Switzerland, FIRMENICH, Switzerland, or International Flavors & Fragrances (IFF), USA. Now, all the experiments turned out negative in the sense that the liquids obtained were not clear but were obviously made up of two distinct phases.

One main object of the present invention is thus to solve the novel technical  
15 problem consisting in the provision of a solution which makes it possible to prepare compositions capable of achieving complete solubilization of perfume concentrates to give an essentially clear liquid, especially for the purpose of preparing perfume compositions.

Another main object of the present invention is to solve the novel technical  
20 problem consisting in the provision of a solution which makes it possible to effect the abovementioned solubilization of perfume concentrates with the aid of solubilizers which are neutral or substantially neutral towards the olfactory properties of said perfume concentrate, especially with a view to preparing perfume compositions of excellent quality.

25 Another main object of the present invention is to solve both the novel technical problems stated above by means of a solution which is free of ethanol or alcohol and which does not use water, ethanol or mixtures thereof as co-solvent.

The invention makes it possible for the first time to solve all these technical problems in a satisfactory and simple manner which can be used on the industrial  
30 and cosmetic scale, especially for the development of ethanol-free or alcohol-free perfume compositions of excellent quality, particularly perfumes and toilet waters.

Thus, according to a first feature, the present invention provides an ethanol-free or alcohol-free composition comprising a hydrofluoro ether, characterized in that it comprises at least one polyacid ester.

35 It has been discovered, surprisingly, that the combination of a hydrofluoro

ether and a polyacid ester, acting as co-solvent, makes it possible to effect a virtually perfect or perfect solubilization of perfume concentrates to afford an essentially clear solution for the manufacture of perfume compositions of excellent quality. Moreover, it has been observed, also surprisingly, that the combination of  
 5 the hydrofluoro ether and the polyacid ester forms a solubilizer which is neutral or essentially neutral from an olfactory point of view towards the perfumes in question.

The hydrofluoro ether component is preferably a perfluorinated component. These compounds are well known to those skilled in the art; they are for example  
 10 of the type described in the document WO 99/11225 and can have the general formula  $C_nH_mF_p-O-C_xH_yF_z$ , in which n is a number ranging from 1 to 12, m is a number ranging from 0 to 25, p is a number ranging from 0 to 11,  $m + p = 2n + 1$ , x is a number from 1 to 12, y is a number from 0 to 25, x is a number from 0 to 11 and  $y + z = 2x + 1$ . In this formula, m and y may not be equal to 0 simultaneously  
 15 and p and z may not be equal to 0 simultaneously, as described in said document to ensure the exactness of the chemical formula.

Some perfluorinated hydrofluoro ether compounds of the above general formula, namely methoxynonafluorobutane, ethoxynonafluorobutane and propoxy-undecafluoropentane, are described in the document WO 99/26600. The  
 20 commercially available methoxyheptafluoropropane can also be used.

The abovementioned polyacid ester is preferably an ester of a hydroxylated or non-hydroxylated polyacid and is also preferably formed with a saturated or unsaturated, linear or branched alcohol having between 1 and 30 carbon atoms, preferably between 1 and 12 carbon atoms, particularly ethanol, isopropanol or 2-ethylhexanol.  
 25

The polyacid preferably has between 3 and 10 carbon atoms. The carbon chain of the polyacid can be linear or branched and saturated or unsaturated with one or more units of unsaturation.

Furthermore, the carbon chain of the polyacids can be substituted by one or  
 30 more hydroxyl groups or can contain one or more ketone groups. The abovementioned hydroxyl groups can be acetylated.

The polyacid esters which can be used to carry out the invention are preferably substantially non-polar.

They can be partial or total esters of the polyacid.

35 Preferably, all the acid groups of the polyacid are esterified.

Preferably, where groups with long carbon chains are involved, these chains are branched, as in the case of the 2-ethylhexyl group, which comprises 8 carbon atoms.

The preferred ester groups are the ethyl, isopropyl and 2-ethylhexyl groups.

5 The polyacids which can be used according to the invention are advantageously selected from:

→ saturated diacids such as:

- malonic acid
- 10 - succinic acid
- glutaric acid
- adipic acid
- pimelic acid
- suberic acid
- 15 - azelaic acid

→ monounsaturated diacids such as:

- fumaric acid
- maleic acid
- 20 - citraconic acid\*
- itaconic acid
- mesaconic acid

\* (branched diacid)

25 → diunsaturated diacids such as:

- muconic acid

→ monohydroxylated diacids such as:

- tartronic acid
- 30 - malic acid
- citramalic acid

→ dihydroxylated diacids such as:

- dihydroxymaleic acid
- 35 - tartaric acid

→ tetrahydroxylated diacids such as:

- dihydroxytartaric acid
- galactaric acid
- glucaric acid

5

→ keto diacids such as:

- mesoxalic acid
- oxalacetic acid
- 2-oxoglutaric acid
- 3-oxoglutaric acid

10

→ diketo diacids such as:

- 2,3-diketoadipic acid

15

→ saturated triacids such as:

- tricarballic acid
- citric acid (monohydroxylated triacid)

20

→ unsaturated triacids such as:

- aconitic acid

The preferred polyacids are citric acid and more particularly adipic acid.

The citric acid esters are preferably triethyl citrate, tri(2-ethylhexyl) citrate and acetyltriethyl citrate. These esters are commercially available.

25

The preferred adipic acid esters are diisopropyl adipate (often called iso-adipate) and di(2-ethylhexyl) adipate. These esters are also commercially available.

30

The proportions by weight of the polyacid ester relative to the hydrofluoro ether component can vary within limits which do not substantially modify the olfactory character of the perfumes. In general, the polyacid ester may represent from 0.1 to 30% by weight, particularly from 1 to 20% by weight, of the hydrofluoro ether component.

35

Provision can also be made for any other complementary component in said composition, particularly a second co-solvent, such as a silicone, or a component for improving the properties of a perfume composition, such as the persistence of

said composition on the skin, an example being a phthalate such as diethyl phthalate. However, silicone is preferred because it simultaneously combines the property of a co-solvent, or solubilizing additive, and the property of persistence.

Preferably, the proportion of silicone will be calculated so as to correspond to about 1 to 20% by weight, based on the weight of the final perfume composition.

Silicones which may be used are a dimethicone or a cyclomethicone, particularly the commercially available pentacyclomethicone, or an organotri-siloxane such as the one described in the PCT document published under the number WO 99/06018 and marketed in particular under the name SILATRIPHENE by RHODIA, France. Advantageously, it will be possible to use volatile silicones, particularly dimethicones marketed by the American company DOW CORNING, such as DC200 Fluid 1 centiStokes or, preferably, DC200 Fluid 0.65 centiStokes.

This composition can also comprise various additives normally used in the preparation of perfume or toilet water compositions, for example UV filters, antioxidants, antioxidants, colors, etc. Such complementary additives will generally be added in a proportion which can range up to 2% by weight of the final composition.

In a first preferred embodiment, the invention provides an ethanol-free or alcohol-free composition comprising a hydrofluoro ether, characterized in that it comprises the following in percentages by weight:

- hydrofluoro ether .....	65 to 85%
- citric acid triester (such as triethyl citrate) .....	4 to 7%
- 2nd co-solvent or solubilizer: silicone such as DC200	
Fluid from DOW CORNING, USA .....	8 to 16%
- perfume concentrate .....	5 to 20%

In a second preferred embodiment, the invention provides an ethanol-free or alcohol-free composition comprising a hydrofluoro ether, characterized in that it comprises the following in percentages by weight:

- hydrofluoro ether .....	65 to 85%
- perfume concentrate .....	5 to 20%
- iso-adipate (or diisopropyl adipate) .....	10 to 20%

Within the framework of the invention, the hydrofluoro ether generally serves to make up the remainder of the composition, but the latter can optionally comprise the various additives normally used in the preparation of the abovementioned perfume or toilet water compositions.

5 It has been observed, within the framework of the invention, that compositions using iso-adipate as the 2nd co-solvent or solubilizing additive generally have a less greasy feel than compositions using the association or combination of citrate and silicone.

10 This represents an important technical advantage, unexpected for those skilled in the art, because the feel of compositions based on iso-adipate is thus closer to that of the conventional alcoholic perfume compositions such as toilet waters and perfumes.

15 According to a second feature, the present invention also covers the use of a polyacid ester, as defined above, in combination with a hydrofluoro ether as solubilizer for a perfume concentrate, especially with a view to preparing perfume compositions such as perfumes or toilet waters.

20 According to a third feature, the present invention also covers an ethanol-free or alcohol-free perfume composition comprising a perfume concentrate and a hydrofluoro ether, characterized in that it also comprises at least one polyacid ester in a sufficient amount to give said composition an essentially clear appearance.

This perfume composition will advantageously comprise from about 65 to about 85% by weight of hydrofluoro ether component, based on the final weight of the perfume composition.

25 According to another advantageous characteristic of this perfume composition, it will comprise from about 1 to about 20% by weight, preferably from 3 to 20% by weight, of abovementioned polyacid ester, based on the final weight of the perfume composition.

30 According to another advantageous characteristic of the perfume composition, it will comprise from about 5 to about 20% by weight of perfume concentrate. In this context, the concentration of perfume concentrate in the case of toilet water will generally be in the order of 5% by weight, based on the final weight of the toilet water. The concentration of perfume concentrate in the case of perfume will generally be between 10 and 20% by weight of the final perfume composition.

35 Thus the perfume composition will preferably consist of a toilet water or a

perfume.

Of course, whether applying to the second feature or the third feature, various modified embodiments result from the first feature. Thus the hydrofluoro ether and the polyacid ester are as defined within the framework of the first feature.

- 5 The same applies to any other complementary component which may be present and which has been described within the framework of the first feature.

Furthermore, within the framework of any one of the features of the invention, the composition can advantageously contain up to 2% by weight, based on the final composition, of any additive normally used in the preparation of  
10 perfume compositions such as perfumes or toilet waters, examples being UV filters, antioxidants, colors, etc.

The process for the preparation of the composition is easy to understand for those skilled in the art. The general procedure will be initially to add the polyacid ester to the perfume concentrate and then to add any complementary compounds,  
15 particularly a second co-solvent, such as a silicone, or a component for improving the properties of the perfume composition, such as the persistence of the composition on the skin, an example being a phthalate mentioned above, any other additives, particularly UV filters, antioxidants or colors, and finally the hydrofluoro ether component, which will preferably be added last and may also generally make  
20 up the remainder of the formulation.

The present invention will now be illustrated with the aid of Examples of ethanol-free or alcohol-free perfume and toilet water compositions with a totally clear appearance or the appearance of a perfect solution; said Examples are given simply by way of illustration and cannot therefore limit the scope of the invention  
25 in any way. Unless indicated otherwise, the percentages in the Examples are given by weight.

Example 1 of an ethanol-free or alcohol-free perfume composition according to the invention

30 This perfume composition has the following formulation:

- ethoxynonafluorobutane .....	68%
- triethyl citrate .....	8%
- commercially available perfume concentrate .....	20%
- diethyl phthalate .....	4%

35 This composition is prepared in the following manner:

The triethyl citrate is first added to the commercially available perfume concentrate and the two are intimately mixed; this is followed by addition of the diethyl phthalate and finally the ethoxynonafluorobutane.

5 The perfume composition prepared in this way, without ethanol or alcohol, is found to have a totally clear appearance or the appearance of a perfect solution, and the olfactory properties of the perfume concentrate are found to be totally preserved.

10 Example 2 of an ethanol-free or alcohol-free perfume composition according to the invention

This perfume composition has the following ingredients:

- methoxynonafluorobutane ..... 71%
- trioctyl citrate ..... 6%
- silicone ref. DC200 Fluid 0.65 cs from DOW CORNING 8%
- 15 - commercially available perfume concentrate ..... 15%

This composition is prepared in a similar manner to that of Example 1, the silicone being added after the citrate.

20 Again it is found that this composition has a totally clear appearance or the appearance of a perfect solution and that the olfactory properties of the perfumes are totally preserved.

Example 3 of an ethanol-free or alcohol-free perfume composition according to the invention

This perfume composition has the following ingredients:

- 25 - methoxyheptafluoropropane ..... 75%
- triethyl citrate ..... 6%
- 50:50 by weight mixture of diethyl phthalate and  
silicone DC200 Fluid 1 cs from DOW CORNING ..... 9%
- commercially available perfume concentrate ..... 10%

30 The mixture is produced in a similar manner to Examples 1 and 2, again giving an ethanol-free or alcohol-free perfume composition with a totally clear appearance or the appearance of a perfect solution, the olfactory properties of the perfumes being preserved.

Example 4: Ethanol-free or alcohol-free toilet water composition according to the invention

This toilet water composition has the following ingredients:

5	- methoxynonafluorobutane .....	82%
	- triethyl citrate .....	3%
	- pentacyclomethicone .....	10%
	- commercially available perfume concentrate .....	5%

This composition is prepared by the same mixing procedure as that described in the previous Examples.

Example 5: Ethanol-free or alcohol-free perfume composition according to the invention

This perfume composition has the following ingredients, again in percentages by weight:

15	- commercially available perfume concentrate, approx. ....	10
	- ethoxynonafluorobutane, approx. ....	77
	- diisopropyl adipate, approx. ....	13

This composition using diisopropyl adipate as an ester co-solvent generally has a less greasy feel than compositions using an association of citrate and silicone, affording the unexpected important technical advantage that the feel of this composition is closer to that of the conventional alcoholic perfume compositions such as toilet waters.

Other modified embodiments of these Examples are well known to those skilled in the art and can include e.g. the incorporation of various other additives normally used in the preparation of perfume compositions such as perfumes or toilet waters, examples being UV filters, antioxidants, colors, etc. Such complementary additives will generally be added in a proportion which can range up to 2% by weight of the final composition.

CLAIMS

1. Ethanol-free or alcohol-free composition comprising a hydrofluoro ether, characterized in that it comprises at least one polyacid ester.
- 5 2. Composition according to claim 1, characterized in that the hydrofluoro ether component is a perfluorinated component, particularly of the general formula  $C_nH_mF_p-O-C_xH_yF_z$ , in which n is a number ranging from 1 to 12, m is a number ranging from 0 to 25, p is a number ranging from 0 to 11,  $m + p = 2n + 1$ , x is a number from 1 to 12, y is a number from 0 to 25, x is a number from 0 to 11 and  $y + z = 2x + 1$ , and in which m and y may not be equal to 0 simultaneously and p and z may not be equal to 0 simultaneously.
- 10 3. Composition according to claim 1 or 2, characterized in that the hydrofluoro ether component is selected from methoxynonafluorobutane, ethoxynonafluorobutane, propoxyundecafluoropentane and methoxyheptafluoropropane.
- 15 4. Composition according to one of the preceding claims, characterized in that the abovementioned polyacid ester is an ester of a hydroxylated or non-hydroxylated polyacid and is preferably formed with a saturated or unsaturated, linear or branched alcohol having between 1 and 30 carbon atoms, preferably
- 20 between 1 and 12 carbon atoms, particularly ethanol, isopropanol or 2-ethylhexanol.
5. Composition according to any one of the preceding claims, characterized in that the polyacid has between 3 and 10 atoms and comprises a linear or branched, saturated or unsaturated carbon chain, it being possible for said carbon chain to be
- 25 substituted by one or more hydroxyl groups or to contain one or more ketone groups, and it being possible for said hydroxyl groups to be acetylated.
6. Composition according to any one of the preceding claims, characterized in that the polyacid is totally esterified with an alcohol as defined in claim 4.
7. Composition according to any one of the preceding claims, characterized in
- 30 that the polyacid is a saturated diacid such as malonic, succinic, glutaric, adipic, pimelic, suberic or azelaic acid, a monounsaturated diacid such as fumaric, maleic, citraconic, itaconic or mesaconic acid, a diunsaturated diacid such as muconic acid, a monohydroxylated diacid such as tartronic, malic or citramalic acid, a dihydroxylated diacid such as dihydroxymaleic or tartaric acid, a tetrahydroxylated
- 35 diacid such as dihydroxytartaric, galactaric or glucaric acid, a keto diacid such as

mesoxalic, oxalacetic, 2-oxoglutaric or 3-oxoglutaric acid, a diketo diacid such as 2,3-diketoadipic acid, a saturated triacid such as tricarballic or citric acid, or an unsaturated triacid such as aconitic acid.

8. Composition according to claim 7, characterized in that the polyacid is citric acid or adipic acid.
9. Composition according to any one of the preceding claims, characterized in that the polyacid ester is a substantially non-polar ester.
10. Composition according to any one of the preceding claims, characterized in that the abovementioned polyacid ester is selected from the group comprising triethyl citrate, tri(2-ethylhexyl) citrate, diisopropyl adipate and di(2-ethylhexyl) adipate.
11. Composition according to any one of the preceding claims, characterized in that the abovementioned polyacid ester represents 0.1 to 30% by weight, particularly from 1 to 20% by weight, of the hydrofluoro ether component.
12. Composition according to any one of the preceding claims, characterized in that it comprises a complementary component, particularly a second co-solvent such as a silicone, or a component for improving the properties of a perfume composition, such as the persistence of said composition on the skin, an example being a phthalate such as diethyl phthalate.
13. Composition according to any one of the preceding claims, characterized in that it comprises a silicone selected in particular from a volatile silicone, a dimethicone, a cyclomethicone, particularly pentacyclomethicone, and an organo-trisiloxane, the silicone preferably representing from 1 to 20% by weight of the composition.
14. Use of a polyacid ester as defined in any one of the preceding claims with a hydrofluoro ether as a solubilizer for a perfume concentrate, especially with a view to preparing a perfume composition such as a perfume or toilet water.
15. Ethanol-free or alcohol-free perfume composition comprising a perfume concentrate and a hydrofluoro ether, characterized in that it also comprises at least one polyacid ester in a sufficient amount to give said composition an essentially clear appearance.
16. Perfume composition according to claim 15, characterized in that the hydrofluoro ether component is present in an amount of about 65 to about 85% by weight, based on the final weight of the perfume composition.
17. Composition according to claim 15 or 16, characterized in that it comprises

from about 1 to about 20% by weight, preferably from 3 to 20% by weight, of polyacid ester, based on the final weight of the perfume composition.

18. Composition according to any one of claims 15 to 17, characterized in that it comprises from about 5 to about 20% by weight of perfume concentrate.

5 19. Composition according to any one of claims 15 to 18, characterized in that it is a toilet water comprising a concentration of perfume concentrate in the order of 5% by weight, based on the final weight of the toilet water.

20. Composition according to any one of claims 15 to 19, characterized in that it is a perfume, the concentration of perfume concentrate being between 10 and  
10 20% by weight of the final perfume composition.

21. Composition according to any one of claims 15 to 20, characterized in that the hydrofluoro ether and/or the polyacid ester are as defined in any one of claims 1 to 11.

22. Composition according to any one of claims 15 to 21, characterized in that  
15 it comprises a complementary component, particularly a second co-solvent such as a silicone, or a component for improving the properties of a perfume composition, such as the persistence of said composition on the skin, an example being a phthalate such as diethyl phthalate.

23. Composition according to any one of claims 15 to 22, characterized in that  
20 it comprises up to 2% by weight, based on the final composition, of at least one complementary additive, for example a UV filter, an antioxidant or a color.

## ABSTRACT

This ethanol-free or alcohol-free composition comprises a hydrofluoro ether and is characterized in that it comprises at least one ester of a polyacid, preferably a hydroxylated polyacid.

This composition can be used in the preparation of perfume compositions such as perfumes and toilet waters.

Docket No.  
71247-0001

# Declaration and Power of Attorney For Patent Application

## English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

### NON-ETHANOLIC COMPOSITION COMPRISING A HYDROFLUOROETHER

the specification of which  
(check one)

☐ is attached hereto.

☒ was filed on AUGUST 25, 2000 ~~as United States Application No. XXXXXX~~ or PCT International  
Application Number PCT/FR00/02384  
and was amended on \_\_\_\_\_

(if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) or Section 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Not Claimed

99/10821

FRANCE

26/08/1999

☐

(Number)

(Country)

(Day/Month/Year Filed)

(Number)

(Country)

(Day/Month/Year Filed)

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(Number)

(Country)

(Day/Month/Year Filed)

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I hereby claim the benefit under 35 U.S.C. Section 119(e) of any United States provisional application(s) listed below:

_____ (Application Serial No.)	_____ (Filing Date)
_____ (Application Serial No.)	_____ (Filing Date)
_____ (Application Serial No.)	_____ (Filing Date)

I hereby claim the benefit under 35 U. S. C. Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112, I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C. F. R., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

_____ (Application Serial No.)	_____ (Filing Date)	_____ (Status) (patented, pending, abandoned)
_____ (Application Serial No.)	_____ (Filing Date)	_____ (Status) (patented, pending, abandoned)
_____ (Application Serial No.)	_____ (Filing Date)	_____ (Status) (patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

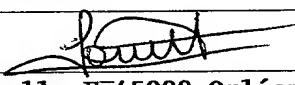
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